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Microwave Dielectric Properties of XM46 and a Surrogate Liquid Propellant

by Robert B. Bossoli

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Microwave Dielectric Properties of XM46 and a Surrogate Liquid Propellant

Robert B. Bossoli
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Abstract

The microwave dielectric properties of liquid propellant (LP) XM46 were determined at room temperature from 2 to 20 GHz using a dielectric probe technique. The dielectric constant (permittivity) of LP was determined in support of possible studies of the feasibility of using microwave energy to preheat LP for more consistent electric ignition in regenerative liquid propellant guns (RLPG). The dielectric properties would also be important in future investigations of the possible development of a safer and more environmentally friendly (over current energetic material-based primers) microwave energy-based LP ignition technique for conventional solid propellants. A surrogate liquid with similar dielectric properties was also developed using water, denatured alcohol, and potassium nitrite (KNO_2), which is not caustic or reactive (like LP) and can be more safely employed in feasibility studies of the use of microwaves in preheating or ignition of LP. This report details the dielectric probe technique for measuring the dielectric constants of liquids such as XM46 LP and the development of the surrogate liquid with matching dielectric properties for use in microwave heating/ignition research and development.

Acknowledgments

The author would like to thank Steven G. Cornelison and Timothy T. Vong for their suggestions and review of this report.

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1. Introduction

U.S. Army researchers and contractors have been exploring the properties and the utilization of a class of materials called liquid propellants (LP) for many years (Irish 1976; Bunte, Vanderhoff, and Donmoyer 1985; Decker et al. 1987; Freedman 1988). They offer the possibility of longer range artillery with faster reloading and lower vulnerability than conventional powder and solid propellants. The U.S. Army Research Laboratory (ARL)* Weapons and Materials Research Directorate (WMRD) has played a key role in the Army's program to develop a 155-mm regenerative liquid propellant gun (RLPG). The basic concept, design, and testing of an electrical initiator for an RLPG can be found in several ARL-WMRD researche reports and papers (DeSpirito, Knapton, and Reeves 1989; DeSpirito, Reeves, and Knapton 1991; DeSpirito and Knapton 1991; Reeves, DeSpirito, and Knapton 1994). Reeves, DeSpirito, and Knapton (1994) investigated the effect of temperature on the electrical ignition of LP. They found that, by keeping the electrical configuration constant, the LP would not ignite in some cases of low (-20° C) and high (40° C and above) temperature extremes. This effect could pose many difficulties in the effort to develop an RLPG suitable for the Army's needs as these temperatures may be experienced in extreme conditions for towed howitzers or if an environmental systems failure occurs in the self-propelled (Paladin/Crusader)-type howitzer.

Vong, Bossoli, and Buffler (1995) have been researching the use of microwaves for preheating stick propellant in tank ammunition in order to increase range and consistency of performance under adverse (low and variable) temperature conditions. By heating the solid propellant to a fixed temperature (say 50° C), an up to 10% increase in velocity with a resultant 5% increase in performance may be achieved (Vong et al. 1996). Microwaves could also be exploited to quickly preheat LP to a standard temperature in order to ensure that the electrical ignition would be reliable and repeatable. LPs are also being investigated for use in several other Army applications, in addition to being a replacement for conventional powder and stick propellants. One possible use is in a detonator role for conventional propellants. An LP-based initiator may offer a safer and improved

* In 1992, the U.S. Army Ballistic Research Laboratory (BRL) reorganized and subsequently became part of ARL.

means of detonating standard propellant charges employed with tank or artillery projectiles. One technique of igniting LP being examined is via lasers (Beyer and Reeves 1997) instead of electrical detonation. Microwaves may also offer the possibility of quickly heating the LP charge to its ignition point in a way that is very reproducible and that could render it insensitive to the temperature of its surroundings. The feasibility of utilizing microwave energy to either preheat or ignite LP is controlled by many factors—one of the most important being its dielectric properties. The interaction characteristics such as absorption and reflection of electromagnetic (EM) radiation are governed by the dielectric properties of a material and its surroundings.

The caustic nature and reactive properties of XM46 can be avoided in initial testing of microwave heating by employing a more benign surrogate. This report details measurement of the dielectric constant of XM46 LP and the development of a surrogate liquid for testing microwave properties such as heating and ignition of LP-like materials. The LP XM46 is a mixture of three liquids. It is 60.8% hydroxyl ammonium nitrate (HAN), 19.2% triethanol ammonium nitrate (TEAN), and 20% water. The density of XM46 is 1.43 g/cm^3, and its electrical conductivity is 0.076 ohm^{-1}-cm^{-1} at room temperature (25° C) (Decker et al. 1987). The large conductivity changes with temperature are evident in results from the investigation by Reeves, DeSpirito, and Knapton (1994), where the initial resistance across the electrode gap in the initiator was found to vary from about 20 ohm at −20° C to 1 ohm above 40° C.

2. Experiment and Theory

2.1 Meaning of Dielectric Constant (Permittivity). The interaction between EM fields and matter is characterized by a substance permittivity and permeability. In our situation, the materials involved do not possess any magnetic characteristics; hence, their relative permeability (relative to free space μ_o) is equal to 1. Therefore, we will only be concerned with our materials relative permittivity, which is equivalent to its dielectric constant. Permittivity is a complex number whose real part represents the ability to store energy and the lossless interaction of the EM field within matter and how the wave behaves at interfaces between different types of materials. Any dissipation

of energy within the material from the EM fields is accounted for in the imaginary part of the permittivity. As stated previously, the dielectric constant is equivalent to relative permittivity, the ratio of a materials permittivity to that of free space, i.e.,

$$\epsilon_r^* = \epsilon^*/\epsilon_o = \epsilon_r' - j\epsilon_r'', \quad (1)$$

where

ϵ_r^* is the complex relative permittivity,

ϵ_o is the permittivity of free space = 8.85×10^{-11} F/m,

ϵ_r' is the storage term, and

ϵ_r'' is the loss term.

2.2 Need for Measurement of the Dielectric Constant. An example of one of the aspects of an EM wave interaction with matter that depends on its dielectric constant is reflection at a surface. Two adjacent materials of differing dielectric constant (e.g., air and a solid surface) can be thought of as an impedance mismatch, and part of the EM wave's energy will be reflected and the rest will be transmitted into the material. Once inside the material, the wavelength and velocity of the EM wave will also be a function of the dielectric constant. If the material displays an imaginary component of the dielectric constant (has loss), there will be attenuation of the EM field magnitude as a function of distance. The energy lost is transferred to the material and usually manifests itself as heat. The imaginary part of the dielectric constant of a material is therefore one of the important factors that will govern the amount of heating that will occur when it is exposed to microwaves inside a cavity or heating chamber.

For a given electric field strength of an EM wave, the power absorbed per unit volume into a material is proportional to ϵ_r'' and the wave's frequency f (Hz) (Von Hippel 1966):

$$P_V = \pi f \epsilon_r'' |E|^2 \text{ (Watt-m}^{-3}\text{)}, \quad (2)$$

where

$|E|$ is the absolute value of the electric field.

Another important EM interaction aspect governed by the dielectric constant is the penetration depth of the waves into the material due to attenuation of the EM waves. The attenuation constant α is (Von Hippel 1966; Blackam, David, and Engelder 1991):

$$\alpha = \pi/\lambda \sqrt{\epsilon_r'} \tan\delta \text{ for } \tan\delta < 1,$$

$$\alpha = \pi (f/c) \sqrt{\epsilon_r'} \tan\delta,$$

and

$$\alpha \approx 10.5 f \sqrt{\epsilon_r'} \tan\delta \text{ (m}^{-1}\text{)}, \quad (3)$$

where

f is the frequency in GHz and

$\tan\delta$ is the loss tangent = ϵ_r''/ϵ_r'.

The attenuation of the power (P_I) of an incident EM wave as it travels through the material is a function of distance (d) and decays exponentially:

$$P(d) = P_I e^{-2\alpha d}. \quad (4)$$

This relationship will be useful in the determination of the optimum design of the microwave injectors and cavity, along with the power necessary to achieve the desired temperatures of the surrogate test material/LP or for achieving ignition of the LP.

2.3 Coaxial Probe Method for Measuring the Dielectric Constant of Materials. The coaxial probe technique consists of placing a specially designed cutoff section of transmission line against a flat surface of a solid material or immersing it into a liquid. The coaxial probe is machined out of inconel (for temperature stability) and glass as the insulating material on termination of the coax line for high temperature and use with caustic chemicals. The EM fields of the probe end fringe into the material (see Figure 1) and the reflected microwave signal (S_{11}) is measured by a microwave network analyzer. A close-up picture of the microwave network analyzer and the dielectric probe is shown in the Figure 2. The probe measures 1.9 cm in diameter at its base, the center of which contains the 0.45-cm diameter glass insulated end of coaxial transmission line. A computer program utilizes the microwave reflectance data to calculate ϵ_r^* over a broad (0.2–20 GHz) frequency range with the permeability (μ_r^*) assumed to be equal to 1 (i.e., nonmagnetic samples). The coaxial probe and software used in the dielectric measurements presented in this report are sold by Hewlett Packard Corporation (HP) (HP 85070B dielectric probe kit) and employed with an HP 8510B network analyzer with the computer program running on an HP series 382 basic workstation controller.

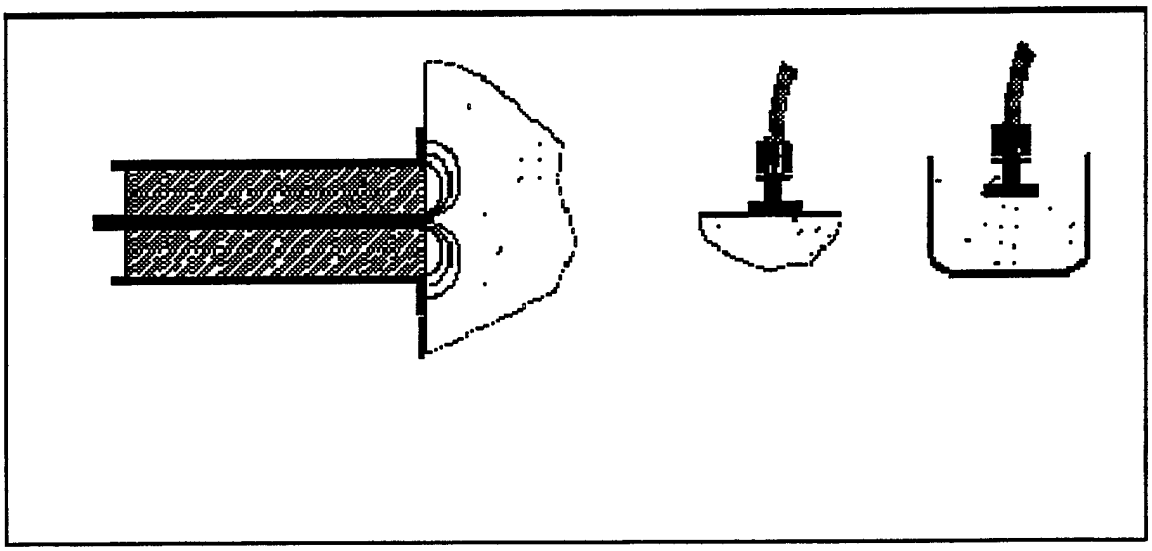


Figure 1. Coaxial Probe Method for Measuring the Dielectric Constant.

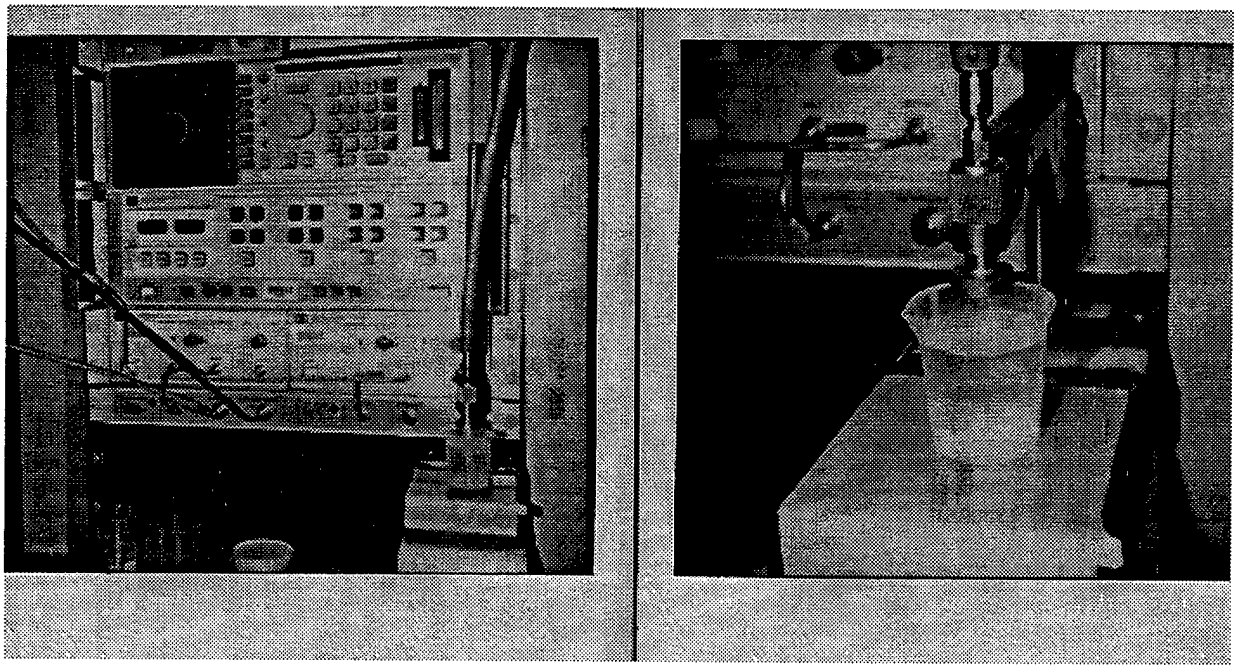


Figure 2. Picture of the Dielectric Probe and Microwave Network Analyzer.

Some of the advantages of the coaxial probe technique for measuring dielectric constant are that it is simple to administer, requiring only a flat surface for a solid material or immersion into a liquid or semiliquid. It is mostly not destructive to a sample and works over a broad band compared to other techniques. For solids, the samples must have a flat measuring surface at least 1 cm diameter and thickness of (Hewlett Packard Corporation 1993)

$$t_{min} > 20/(\epsilon_r')^{1/2} \text{ (mm)}, \tag{5}$$

typically 1 cm in thickness or greater. The HP 85070B dielectric probe allows for sample measurements from −40 to +200° C. The major drawback to this method is that it has limited accuracy for measuring ϵ_r^* (± 5% at best) and has limited resolution on low-loss samples. Accuracy in tanδ is ± 0.05, with the minimum recommended tanδ of 0.05 for values of $\epsilon_r' > 5$ (Hewlett Packard Corporation 1993). The probe and network analyzer measures the reflection parameters, which are used in the computer program to calculate the dielectric constant. Sources of error in this procedure come from noise and residual systematic errors not handled by the calibration procedure

discussed in the next section. The larger "dielectric errors" come from the probe model accuracy (3–5%) and uncertainty of the dielectric characterization of the calibration (reference) standards (such as water).

The simple admittance model for the probe as explained in the operating manual for the probe (Hewlett Packard Corporation 1993) is shown in Figure 3. The coaxial probe can be modeled by a circuit whose total admittance is composed of a pure capacitance (Ca), which can be related to ϵ_r' and, a conductance (Co), which is related to ϵ_r''. An additional term (G_r-conductance) represents radiation losses of the probe configuration (which become larger at high frequencies) and is a major element in limiting the sensitivity in determining ϵ_r'' using this method.

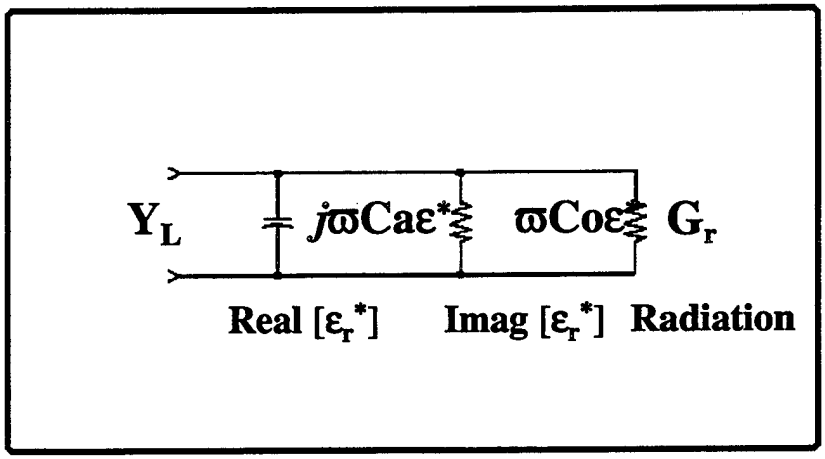


Figure 3. Circuit Model for the Dielectric Probe.

2.4 Calibration and Errors in Dielectric Probe Measurements. As stated previously, an accurate measurement of a sample's reflection parameter with the probe is necessary to calculate its dielectric constant. In performing the reflection measurement, calibration of the probe using three well-known standards allows for the removal of the systematic errors of directivity, tracking, and source match. In network analysis, these systematic errors are introduced in the measurement equipment. Directivity errors refer to the leakage from reflections from unwanted paths (i.e., wrong

direction) in the internal directional couplers in the system test set. The tracking or frequency response errors are due to slight differences in the phase and magnitude of the signals in the reference and test channels as a function of frequency. Finally, source match errors result from differences in impedance along the measurement paths (mismatches) due to connectors, cables, and the probe, which result in unwanted reflections. The three known standards typically employed for calibration are air, a short circuit, and deionized water. Using these standards (applied at the probe surface during calibration) and an error model, taking into account what the network analyzer system should measure for each standard, the dielectric probe computer program sets up the network analyzer such that these systematic errors are removed from the measured data. Other errors that cannot be corrected and can cause inaccuracy in the measurements are flexing of the cable connecting the probe to the network analyzer and temperature changes in the cable (not stabilized as a function of time). The flatness of the measurement surface on solid materials can also lead to significant errors if there are any minute gaps between the probe and the sample. In measurements of the liquids reported here, this error is not present. The sample must also appear to be "infinite" to the probe with the minimum thickness given by the previous equation.

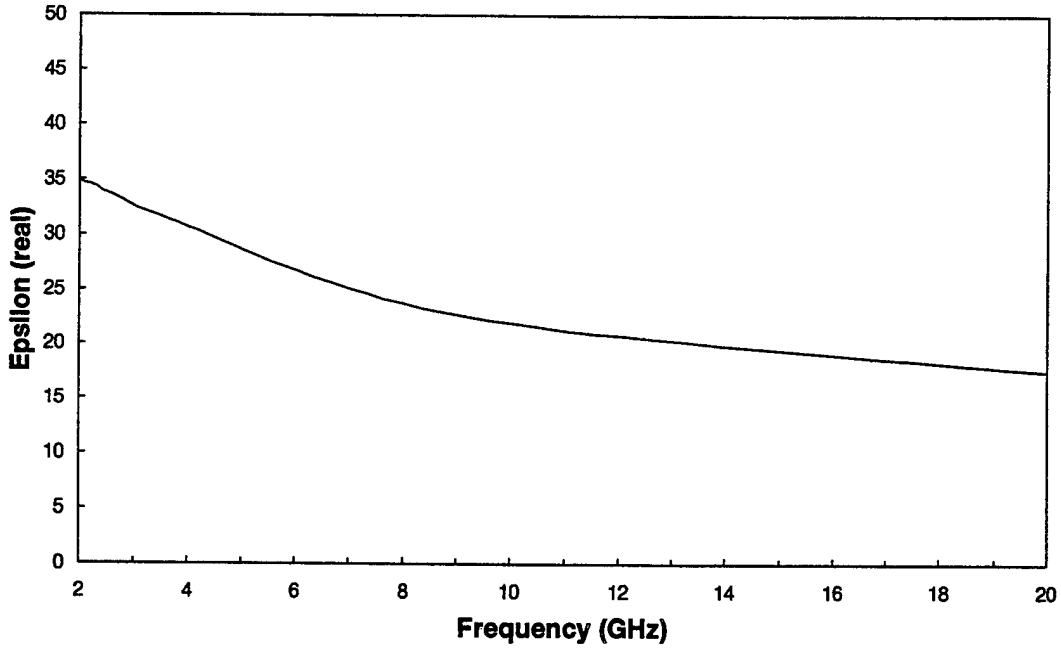
As stated earlier, one of the drawbacks of the coaxial probe technique is its limited accuracy, especially for low-loss materials. The best resolution of tanδ is ± 0.05. The probe's precision worsens at lower frequencies; for our 2–3-GHz frequency range of interest for microwave heating, the best accuracy in ϵ_r' is approximately 10% for a dielectric constant of 5 and ± 7% for a dielectric constant around 20 (Hewlett Packard Corporation 1993). This does not include any inaccuracies caused by incomplete contact between the probe and the sample surface and those caused by cable instability (movement and thermal).

As seen in the following sections, the dielectric probe provided measurement of the complex dielectric constant for an XM46 LP sample and helped guide the design of a surrogate liquid material for possible utilization in the testing of microwave-heating cavities and ignition chambers for LP-type materials.

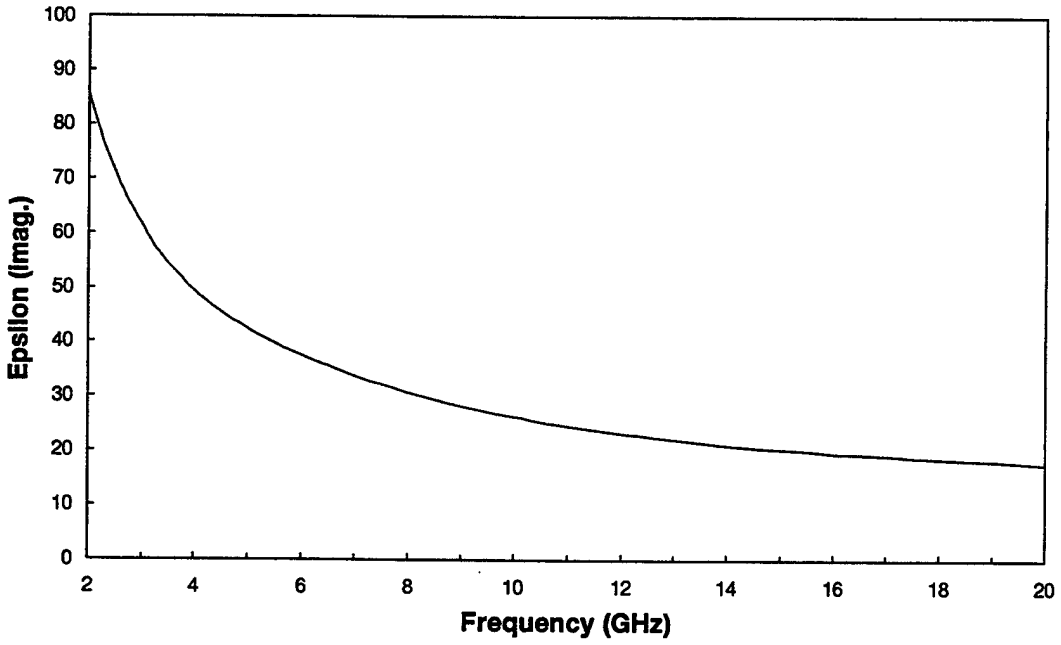
3. Results and Discussion

3.1 Dielectric Properties of XM46. Figure 4 shows the real and imaginary parts of the dielectric constant of XM46 from 2 to 20 GHz. The large 1/f dependence of the slope of the ϵ_r'' curve (Figure 4b) is characteristic of materials where ionic conductivity is present (Hewlett Packard Corporation 1992). Ionic conductivity is usually the main mechanism for the large losses in the liquids at lower frequencies. Ionic conductivity however contributes only to losses (i.e., the imaginary part of the dielectric constant). The fairly large value of the real part of the dielectric constant (ϵ_r') (Figure 4a) is probably due to water a component (20%) of XM46 LP. Pure distilled water has a dipolar loss mechanism that has a characteristic resonant (or relaxation) frequency in the microwave region (Hewlett Packard Corporation 1992). Figures 5a and b show the real and imaginary parts of the dielectric constant for pure distilled water. The loss mechanisms giving rise to a large ϵ_r'' in this case are rotation and alignment of the water molecules dipole moment to the electric field of the microwaves. In Figure 5b, ϵ_r'' is seen to slowly increase with the microwave frequency with a peak corresponding to a relaxation frequency just above 20 GHz. Above this frequency, ϵ_r'' begins to fall off, as the electric field oscillation is too fast for the water dipoles to keep up with and, hence, the loss decreases. This polarization effect in water also leads to a large real part of the dielectric constant (ϵ_r'), which, as shown in Figure 5a, is much larger than that of XM46. In order to construct a surrogate liquid with the same dielectric properties as XM46, a good starting point would be to utilize water as one of the components (like LP XM46). Other components will be looked at to safely add some ionic conductivity to water to increase the loss term or imaginary part of the dielectric constant. Comparing Figures 4a and 5a shows that the real part of the dielectric constant of XM46 is lower by about a factor of 2 than that of distilled water over the 2–20-GHz range. Other components are needed to reduce the real part of the dielectric constant of distilled water in the range of XM46 LP.

3.2 Dielectric Properties of Surrogate Solutions. Ethyl alcohol is another dipolar liquid, but with real and imaginary parts of the dielectric constant smaller than water over the 2–20-GHz frequency range (see Figure 6). A mixture of distilled water and ethanol (denatured) should be able

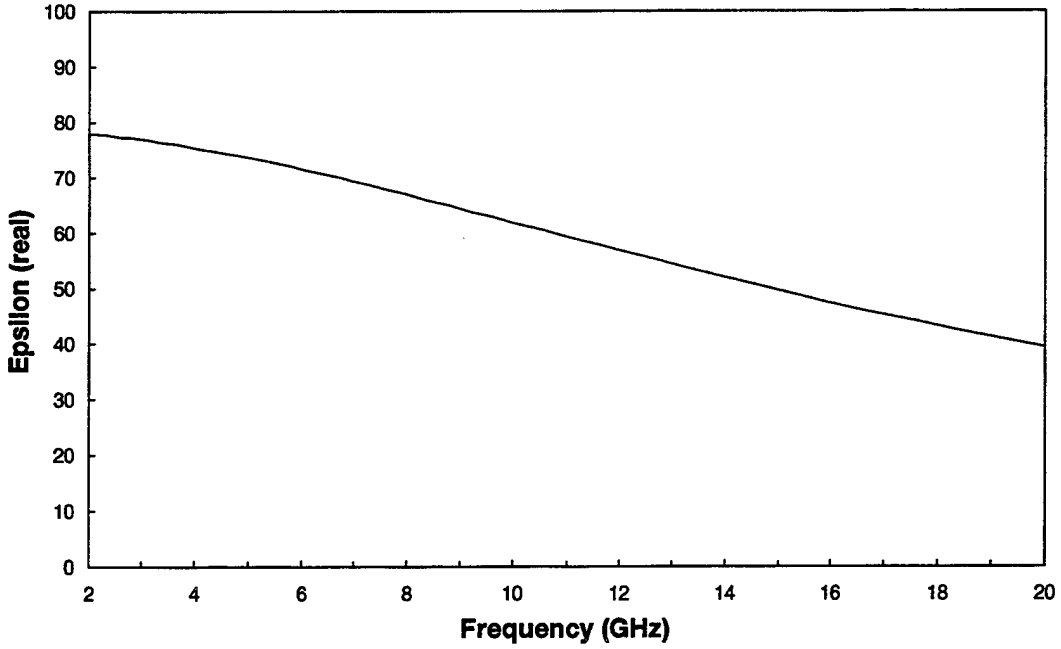


(a) Real.

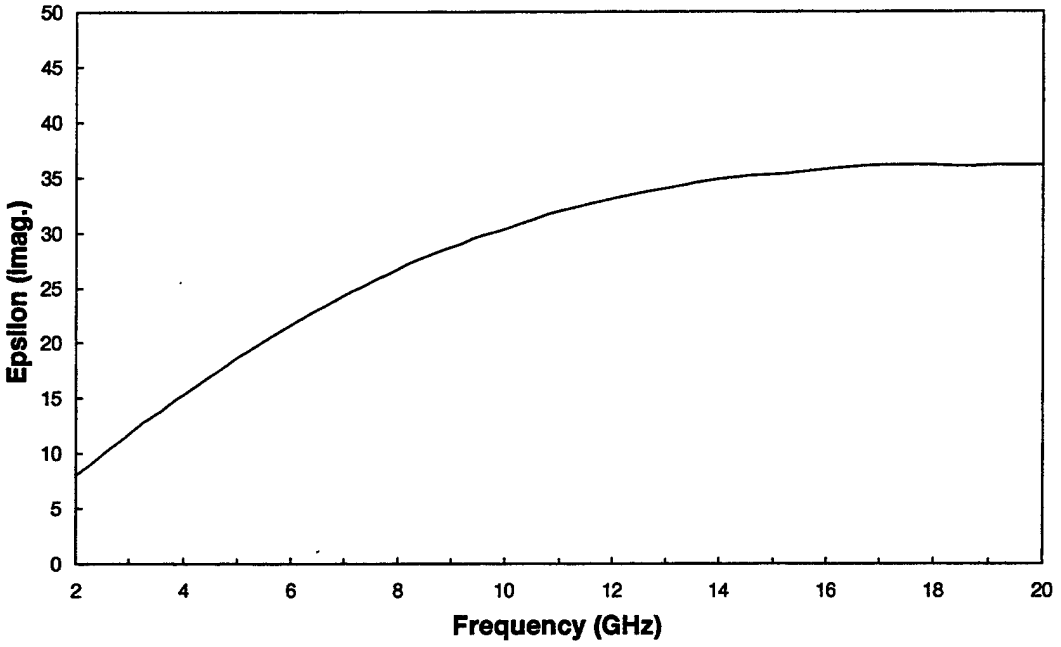


(b) Imaginary.

Figure 4. Real and Imaginary Parts of the Dielectric Constant of XM46 LP.

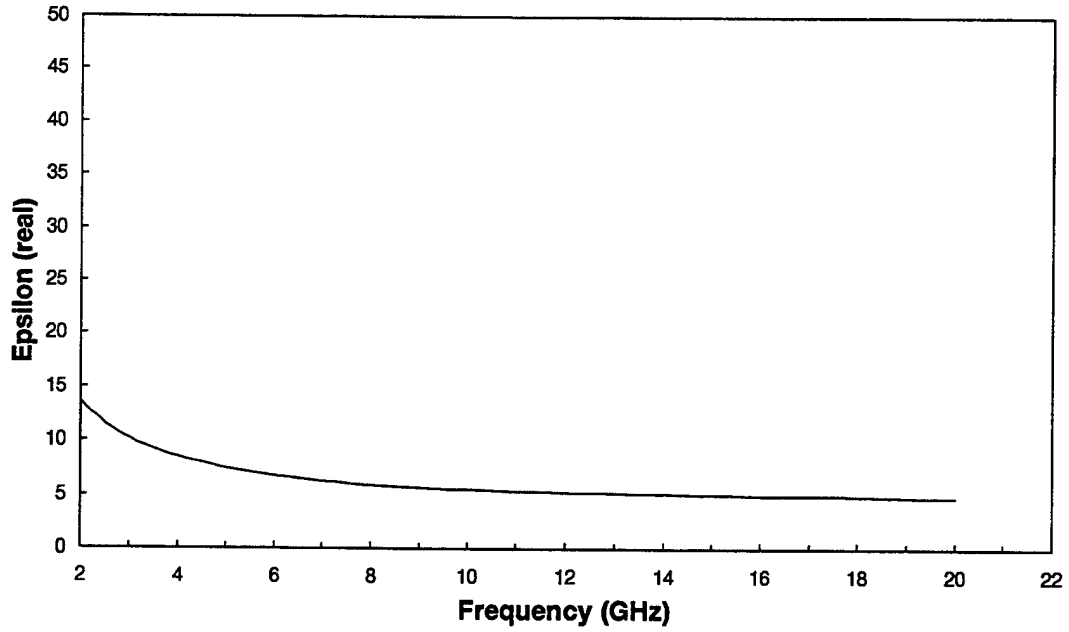


(a) Real.

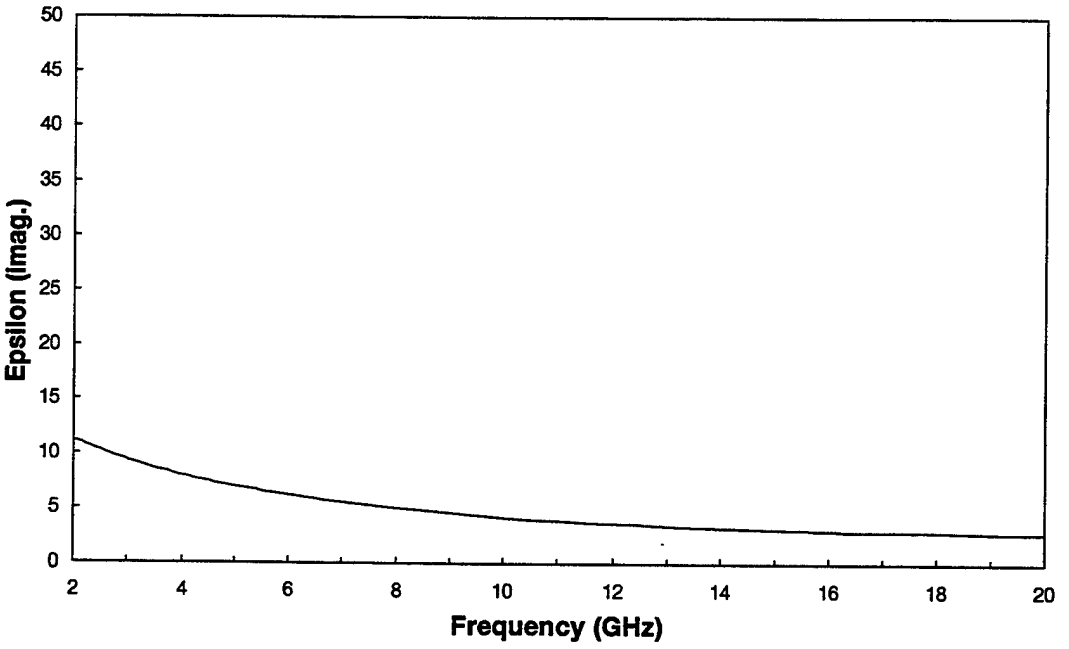


(b) Imaginary.

Figure 5. Real and Imaginary Parts of the Dielectric Constant of Distilled Water.



(a) Real.



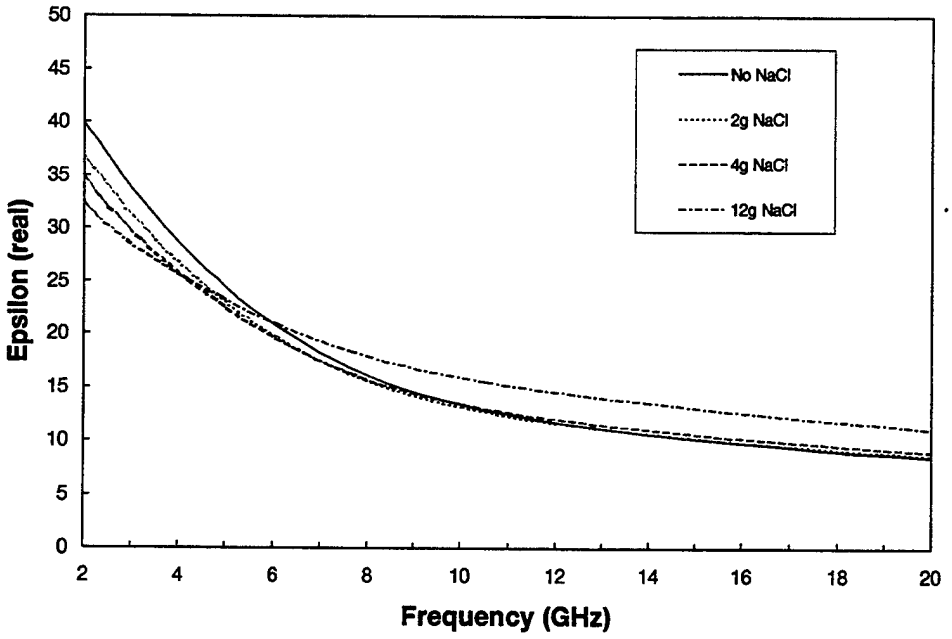
(b) Imaginary.

Figure 6. Real and Imaginary Parts of the Dielectric Constant of Denatured Ethanol.

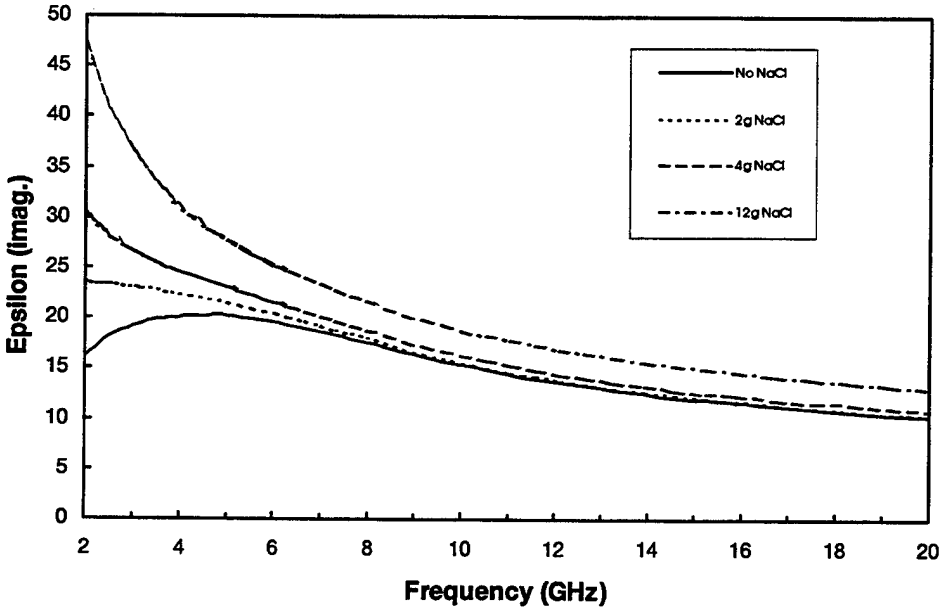
to come close to reproducing the real part of the dielectric constant. Figure 7 shows the real and imaginary parts of the dielectric constant measured with the dielectric probe for a 65/35 (100 cm^3) mixture of denatured alcohol and distilled water. It also shows the effect of adding an ionic conducting component salt (NaCl) to the mixture. All samples were prepared and measured at room temperature (~23° C), and a magnetic stirrer was used to mix in the ionic salt component. The addition of salt to the mixture is seen to greatly increase ϵ_r'' but only slightly change ϵ_r', causing it to drop slightly. However the increase in the imaginary part (loss) of the dielectric constant is not large enough be comparable to that of XM46 for the 12 g of NaCl in the 100 cm^3 of the mixture. The 12 g of NaCl was over the maximum soluble amount (i.e., not all of the salt was dissolved into the solution).

Another ionic material that would be more soluble in water and ethanol needed to be found. Potassium nitrite (KNO_2) is an ionic salt with a much higher solubility (281 g/100 cm^3 water) than NaCl (35.7 g/100 cm^3 water) and was added to a 65/35 solution (100 cm^3) of ethanol and distilled water. Figure 8 shows the real and imaginary dielectric constant of the solution with 0, 10, and 28 g of KNO_2 added. Comparing the results with Figure 4 for XM46, the real and imaginary parts of the solution with 28 g of KNO_2 come close but are still slightly smaller. In addition, with this larger amount of KNO_2, ϵ_r' has fallen below 35, the XM46 value at 2 GHz. A different starting solution of ethanol and distilled water was tried next (larger percentage of water) to compensate for this effect.

A 50/50 solution (100 cm^3) of ethanol and distilled water was prepared, and 25-, 28-, and 30-g amounts of KNO_2 were added and measured with the dielectric probe. The results, along with the pure solution (no KNO_2), are shown in Figure 9. The mixture with 30 g of KNO_2 in the 50/50 cm^3 ethanol-water mixture comes very close to matching the dielectric properties (ϵ_r' and ϵ_r'') of the XM46 LP. The dielectric probe data for XM46 and 30 g of KNO_2 in the 50/50 mixture of water and alcohol are displayed together in Figure 10. The imaginary part of the dielectric constant (Figure 10b) of the ethanol-water-KNO_2 solution has an excellent match to XM46 LP over the whole 2–20-GHz frequency range. The data for ϵ_r' in Figure 10a show that the surrogate mixture comes

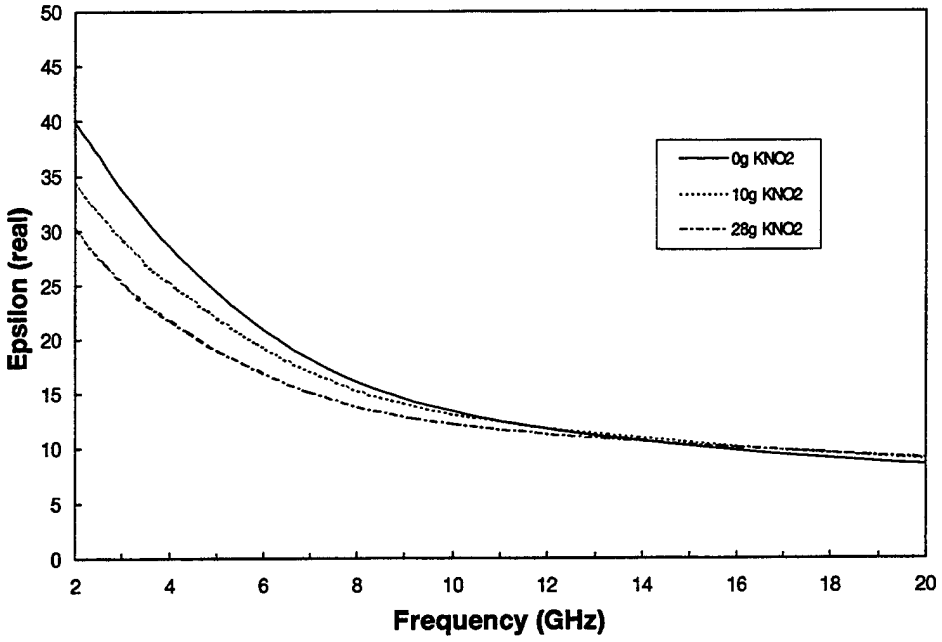


(a) Real.

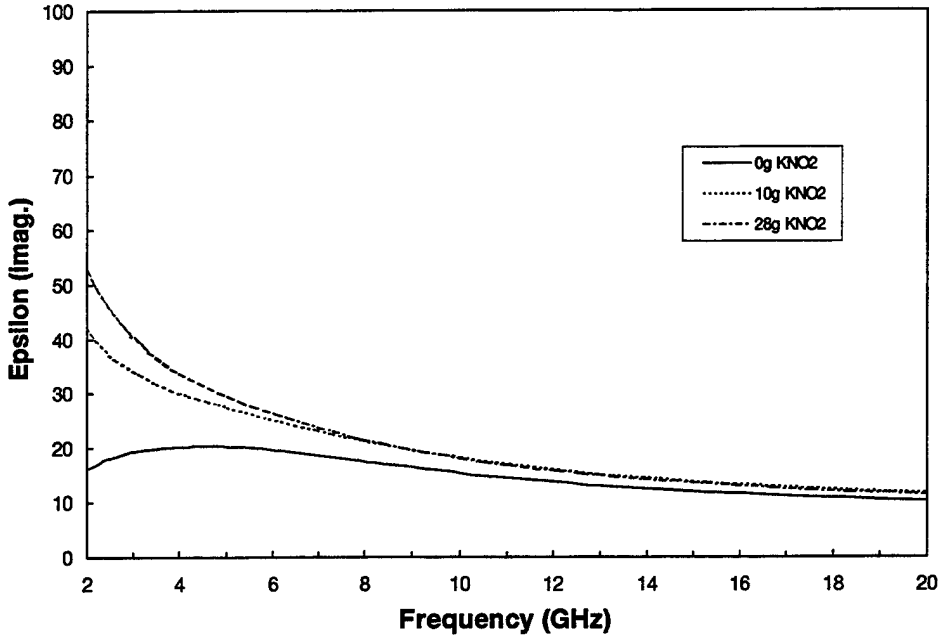


(b) Imaginary.

Figure 7. Real and Imaginary Parts of the Dielectric Constant of 65/35 Mixture of Ethanol and Distilled Water with 0, 2, 4, and 12 g of NaCl Added.

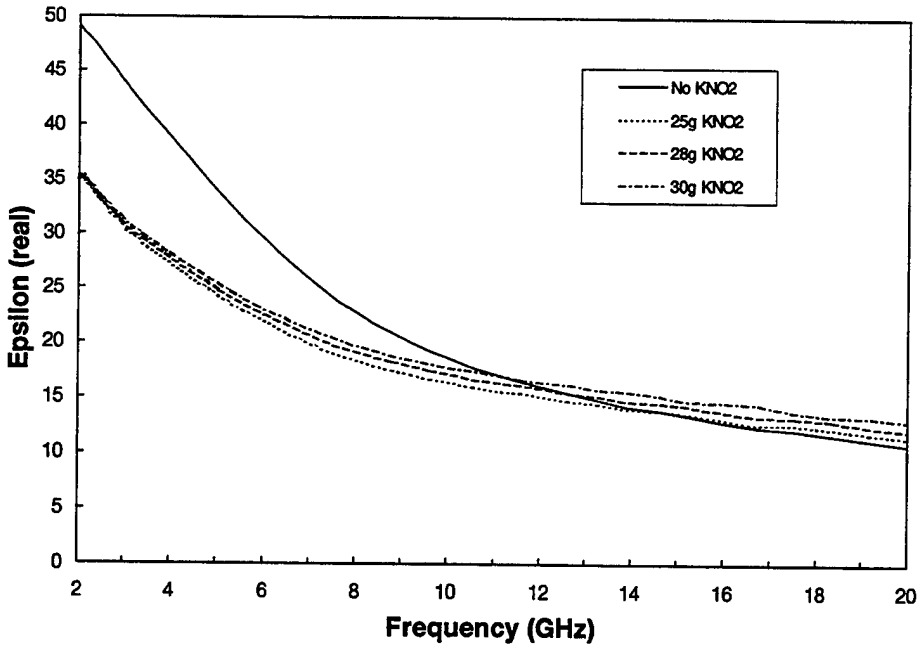


(a) Real.

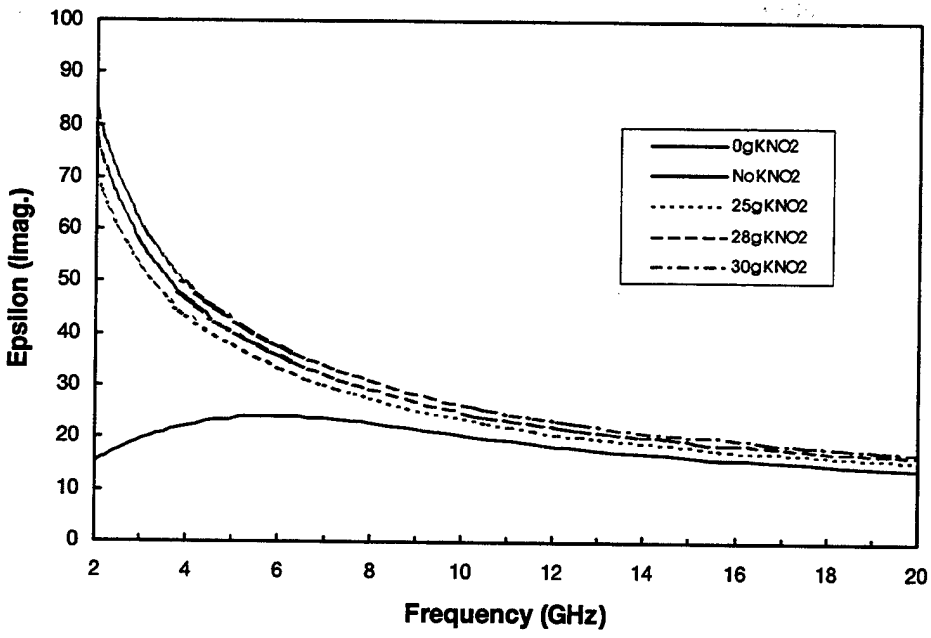


(b) Imaginary.

Figure 8. Real and Imaginary Parts of the Dielectric Constant of 65/35 Mixture of Ethanol and Distilled Water With 0, 10, and 28 g of KNO_2 Added.

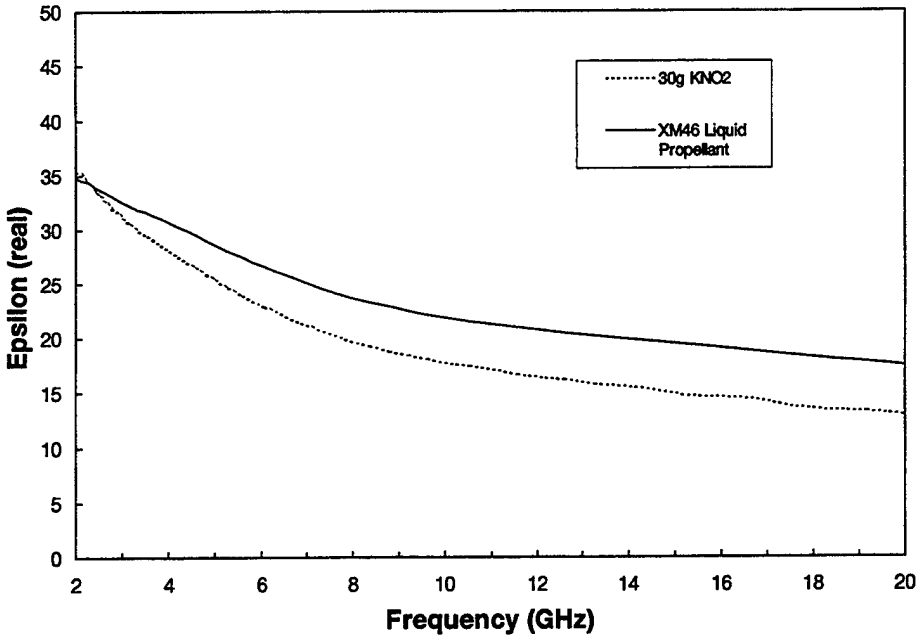


(a) Real.

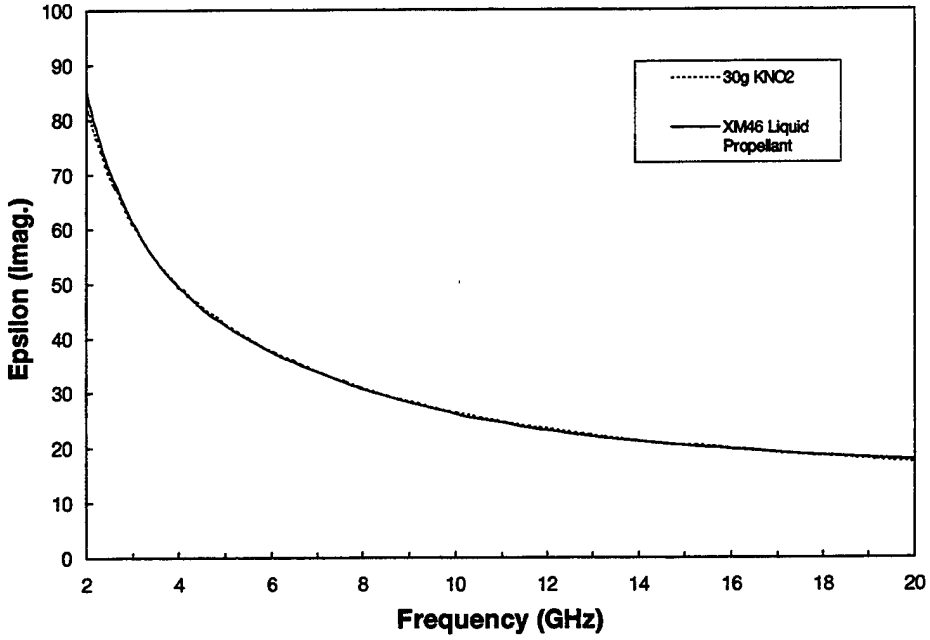


(b) Imaginary.

Figure 9. Real and Imaginary Parts of the Dielectric Constant of 50/50 Mixture of Ethanol and Distilled Water With 0, 25, 28, and 30 g of KNO_2 Added.



(a) Real.



(b) Imaginary.

Figure 10. Comparison of the Real and Imaginary Parts of the Dielectric Constant of Ethanol-Water-KNO_2 Solution and XM46 LP.

close to matching the XM46 in the standard operating range (2–3 GHz) for microwave ovens and generators utilized in heating liquids and drying moist materials. If a different operating frequency for the microwave heating is employed, a slightly different ratio of the three components could be made to match ϵ_r^* for XM46 or other types of LPs with similar chemical compositions.

4. Conclusions

The dielectric properties of XM46 LP have been investigated over the 2–20-GHz microwave frequency range. LP exhibits high real and imaginary parts of the dielectric constant due to its conductive and dipolar liquid properties. This will make microwave penetration (coupling into) and even heating of the material difficult but doable in a properly constructed cavity. The microwaves will efficiently heat the liquid due to its high loss factor (ϵ_r''). This high absorption factor also increases the feasibility of obtaining ignition of LP via high power microwaves.

A less volatile and caustic dielectric surrogate for XM46 LP has been concocted and matches ϵ_r'' very well over the 2–20-GHz frequency band. The real part of the dielectric constant (ϵ_r') of the surrogate LP closely matches XM46 in the important 2–3-GHz regime, where microwave-heating and -drying equipment usually operate. This surrogate liquid can be employed for safe initial testing of the designs for any microwave-heating chamber for LP or microwave-based LP ignition device.

5. References

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13. ABSTRACT *(Maximum 200 words)*

The microwave dielectric properties of liquid propellant (LP) XM46 were determined at room temperature from 2 to 20 GHz using a dielectric probe technique. The dielectric constant (permittivity) of LP was determined in support of possible studies of the feasibility of using microwave energy to preheat LP for more consistent electric ignition in regenerative liquid propellant guns (RLPG). The dielectric properties would also be important in future investigations of the possible development of a safer and more environmentally friendly (over current energetic material-based primers) microwave energy-based LP ignition technique for conventional solid propellants. A surrogate liquid with similar dielectric properties was also developed using water, denatured alcohol, and potassium nitrite (KNO_2), which is not caustic or reactive (like LP) and can be more safely employed in feasibility studies of the use of microwaves in preheating or ignition of LP. This report details the dielectric probe technique for measuring the dielectric constants of liquids such as XM46 LP and the development of the surrogate liquid with matching dielectric properties for use in microwave heating/ignition research and development.

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